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LOW TEMPERATURE THERMAL EXPANSION OF STRUCTURAL METALS

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NASA

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ABSTRACT

Thermal expansion determinations were made on two aluminum alloys and three stainless steel alloys in the temperature range from 4° to 300°K. The experimental apparatus is described, and the analytical procedures which were required to reduce the data accurately to a usable form are discussed. The data are presented in both tabular and graphical form, and both linear coefficient of expansion and total percentage of expansion as a function of temperature are given.

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PROPULSION AND VEHICLE ENGINEERING LABORATORY
RESEARCH AND DEVELOPMENT OPERATIONS

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LOW TEMPERATURE THERMAL EXPANSION OF STRUCTURAL METALS

SUMMARY

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An apparatus for the determination of low temperature linear thermal expansion of metals and plastics in the temperature range from 4° to 300°K is described. The apparatus was developed by Midwest Research Institute (MRI) on Contract NAS8-835, under the technical direction of the Materials Division, and was subsequently delivered to MSFC and installed in the thermal properties laboratory of this division. The thermal expansion determinations described in this report were made to provide useful engineering data and qualification and calibration of the apparatus.

Modifications made to the apparatus to permit automatic data recording are discussed, and the data reduction process resulting from these modifications are discussed in detail. It is shown that data points, taken under equilibrium conditions, are possible at intervals of 10°K, which produces an extremely smooth and accurate data curve.

Determinations were made of the linear coefficient of thermal expansion for 321, 347, and 355 stainless steels, and 2017-T4, 6061-T6, and 6061-T651 aluminum alloys in the temperature range from 10° to 300°K. The calibration and qualification run was made on 347 stainless steel since MRI had made calibration runs on the same material. Comparison of the data from this run with the MRI data indicates excellent agreement.

Data from all determinations are presented in both tabular and graphical form and both linear coefficient of expansion and total percentage expansion are given.

The apparatus is shown to be capable of determining the linear coefficient of thermal expansion in the temperature range from 10° to 300°K with an accuracy of $\pm$2.0 percent.

INTRODUCTION

Author

The extensive use of cryogenic propellants in most large launch vehicle systems has created a need for accurate measurement of the thermal properties of structural materials down to the temperature of liquid

hydrogen (20°K). The large propellant tanks of these vehicles serve not only as propellant containers but also as an integral part of the structure. Thus, the low temperature thermal expansion of the tank materials is important to the vehicle designer. The propellant tanks will be cooled from ambient to cryogenic temperature when filled, thus resulting in overall contraction; however, they still must mate properly with the main thrust structure, which is still at ambient temperature, without producing undue stress at the connections.

As an example, the Saturn V liquid oxygen tank is 1,000 centimeters in diameter, with a circumference of 3,140 centimeters. The tank is constructed of aluminum and, during cooldown from ambient temperature (300°K) to liquid oxygen temperature (92°K), undergoes a thermal contraction of 11.6 centimeters, which must be absorbed by the thrust and load structures. It is necessary for the designer to know the thermal expansion (or contraction) for the various alloys of aluminum or other structural materials which might be used to provide an allowance for this contraction.

To provide this information, a low temperature thermal expansion apparatus was developed by MRI on contract NAS8-835 under the technical supervision of the Materials Division. This apparatus subsequently was delivered and installed in the thermal properties laboratory of this division. The thermal expansion determinations to be described were made to provide useful engineering design data and qualification and calibration data for this apparatus.

THERMAL EXPANSION

At absolute zero (0°K), when the atoms in a solid are at rest (neglecting the quantum-mechanical zero point energy), the actual volume of the solid will be that for which the energy is a minimum (ref. 1). At any other temperature (T), when the atoms are not at rest but are vibrating about their equilibrium positions, the volume of the solid is greater because of the resulting thermal expansion.

If the expansion in only one direction (linear expansion) is considered, a small change in temperature (dT) produces a small change in length (dL) and allows the definition of a linear coefficient of thermal expansion, alpha,

$$\alpha = \frac{1}{L}\frac{dL}{dT} \qquad \text{(equation 1 (ref. 2))}$$

where L is the initial length;
dL is the change in length;
dT is the change in temperature.

Thus, the coefficient is actually the instantaneous slope of the line resulting from a plot of sample length versus temperature.

From this relation, an expression relating the sample length to initial length at any temperature may be obtained. Since

$$\alpha = \frac{1}{L} \frac{dL}{dT}$$

separating variables

$$\frac{dL}{L} = \alpha \, dT$$

integrating

$$\ln L = \alpha T + C$$

taking antilogarithms of both sides

$$L = \exp C \exp (\alpha T) = D \exp (\alpha T)$$

evaluating the constant D for the initial conditions that at T = 0, L = Lo, the length at temperature, T,

$$L_T = L_o \exp \alpha T \approx L_o (1 + \alpha T) \qquad \text{(equation 2)}$$

The proper use of this relation requires that the coefficient be constant within the temperature range of interest or that it be averaged over the temperature range from T = 0 to T = T,

$$\alpha \text{ average} = \frac{L_2 - L_1}{L_1 (T_2 - T_1)} \qquad \text{(equation 3)}$$

where L_2 and L_1 are the lengths at temperature T_2 and T_1 respectively. This quantity is the average value of the slope of the length versus temperature plot. It should be noted that α average is not the true coefficient of linear thermal expansion and can differ from the true value by an appreciable amount if the temperature span is wide.

In an isotropic substance, the coefficient of volume expansion, beta, is simply three times alpha. However, if a material is anisotropic, the linear coefficient is found to vary as the line along which it is observed takes different orientations with respect to the crystal axes (ref. 3). In general, it is possible to choose three directions orthogonal (not necessarily the crystallographic axes) such that the linear coefficient of expansion of the three axes describes the expansion of the crystal as a whole.

In many solids, hysteresis is observed (ref. 2). When these solids are heated and subsequently cooled, they do not regain their original dimensions. This is expected in polycrystalline materials because of the variation in size of the individual grains and their purely random packing (ref. 3). Large alterations in the size and arrangement of the crystalline grains may be produced during a heating-cooling cycle and this is not a reversible process. It is often helpful to make hardness determinations on a sample before and after making a test run to insure against misinterpretation of results caused by this effect (ref. 4).

EXPERIMENTAL APPARATUS

The apparatus used has been fully described in the open literature (ref. 5), and only a brief resume of the basic operation will be included in this report. The basic measuring arrangement is shown in block form in FIG 1 and in detail in FIG 2 and 3. It consists of five major elements: (1) measuring head, (2) interferometer, (3) photomultiplier detector, (4) photometer amplifier, and (5) recorder and readout.

The measuring head is suspended in a deep double-wall, silvered-glass dewar containing the appropriate cryogenic fluid. Sample temperature is controlled by a small heater (10 turns of 28 gauge constantan wire wrapped around the sample holder), and the sample expansion is transmitted to the lower Fizeau interferometer plate through a quartz rod driving a precision metal piston. Movement of the lower plate with respect to the fixed upper Fizeau plate (caused by expansion or contraction of the specimen) results in the movement of the interference fringe pattern in direct proportion to the movement of the specimen. A portion of the near field pattern is focused on the first dynode of a six-stage electron multiplier photodetector by means of appropriate optics aligned axially with the interferometer collimator. As the field pattern changes from a bright fringe to a dark fringe, the change in intensity is detected by the electron multiplier, amplified in the photometer amplifier, and displayed on a potentiometric recorder. The apparatus was modified at this Center by equipping the recorder with a cam-driven microswitch which converts the voltage swing to drive a digital counter. The counter then reads the total number of fringes directly; thus, the expansion of the sample is determined easily.

Temperature measurement is accomplished by copper constantan thermocouples using a 32°F reference. The thermocouple potentials are read with a K-3 potentiometer utilizing a Zener stabilized working voltage source and are converted to equivalent temperature readings by the use of NBS standard tables (ref. 6).

MEASUREMENT TECHNIQUE

Cryogenic fluid is transferred into the dewar, and the sample temperature is monitored until the sample reaches equilibrium, as indicated by the temperature and by the fringe counter (stationary fringe pattern). Then, voltage is applied to the sample heater until the fringe pattern begins to move. Heater voltage is kept constant until the sample again reaches equilibrium as indicated by both the temperature and the fringe pattern. The heater voltage is increased by another small increment, and this technique is continued throughout the run. Temperature and total fringe counts at that temperature are recorded at intervals of approximately ten degrees throughout the run.

DATA REDUCTION

Since the passage of one fringe represents a change of one-half wavelength in path distance between the two interferometer plates, the expansion or change in sample length is

$$d = N \lambda/2$$

where

d is the change in length
N is the number of fringes
λ is the wavelength

The interferometer is operating from the 5461 Angstrom green line of mercury (filtered) so each fringe represents a length change of 2730 Angstroms, or 2.73×10^{-5} centimeters. The total number of fringes at a given temperature and the sample initial length (at ambient temperature) is known; therefore, a plot of sample length as a function of temperature may be obtained. The linear coefficient of thermal expansion may be obtained directly from this plot by taking a uniform increment of temperature, measuring the change in length over this temperature span, and

$$\alpha = \frac{1}{L_1} \quad \frac{L_2 - L_1}{T_2 - T_1} \qquad \text{(equation 4 (ref. 2))}$$

where α is the linear coefficient of the expansion in cm/cm °K;

L_1 is the length at temperature T_1;
L_2 is the length at temperature T_2;
T_1 is the initial temperature;
T_2 is the final temperature.

This, of course, is the value of alpha averaged over the temperature span from T_1 to T_2 and not the point slope; however, if the temperature span is not too wide, it is a close approximation to the true value.

The plot of length as a function of temperature also served as a basis for the total percentage expansion data in which

$$\text{percent expansion} = \frac{L_T - L_{300}}{L_{300}\ (300\text{-}T)} \qquad \text{(equation 5 (ref. 7))}$$

where L_T is the length at temperature (T) and L_{300} is the length at 300°K.

INTERFEROMETRIC TECHNIQUE ERRORS

There were three primary sources of error in the interferometric method of determining thermal expansion as originally proposed and built by Fizeau (ref. 2). These were errors caused by (1) the change in wavelength resulting from the change in index of refraction of the air between the plates as the temperature and pressure change (ref. 8), (2) the increase in optical path length resulting from the thermal expansion of the interferometer plates (ref. 9), and (3) tilting of the interferometer plates by nonuniform expansion of the samples (ref. 8 and 10). The instrument designed by MRI has eliminated these sources of error by removing the interferometer plates from the heated (or cooled) area and using a single sample with a precision fitted metal piston to assure continuous alignment (ref. 5), as shown in FIG 2. This approach had been used before (ref. 10), but, while eliminating the three sources of error, it had introduced an additional larger error. This was due to the long quartz rod which was used to transmit the specimen expansion to the interferometer plates. Due to the length of this rod (despite the low thermal expansion of quartz), the total change in length of the rod was appreciable. MRI compensated for this effect by supporting the specimen on the bottom of a quartz tube enclosing the measuring head. As temperature increases, the rod will get longer and move the measuring piston up; the outer quartz tube also will increase in length, downward, which will lower the sample and quartz rod. If the temperature of the quartz rod and the quartz tube are the same (or, more accurately, if the thermal gradients are the same) and the two are equal in length, the total system is compensated, and the net movement is zero. In this apparatus, a small difference in length does exist between the quartz rod and the quartz tube. However, heat is applied directly to the sample by an enclosing cylinder, and the only heat input to the quartz rod and tube is by conduction from the sample through a small contact area. Additionally, the outer tube is in optical contact with the surrounding nitrogen dewar and encloses the quartz rod, which should ensure the maintenance of an equal thermal gradient. For these reasons, no quartz correction terms were applied.

DISCUSSION OF DATA

Determinations were made of the linear coefficient of thermal expansion for six materials, three 300 series stainless steel samples and three aluminum samples. Two of the aluminum samples differed only in heat treatment. They were deliberately chosen to illustrate the change in expansion coefficient produced by heat treatment.

The calibration and qualification run was made with a sample of 347 stainless steel since MRI had made calibration runs on the same material when the apparatus was installed in their laboratories. Figure 4 is a plot of linear coefficient of thermal expansion as a function of temperature for 347 stainless steel. Figure 5 is a plot of percent expansion as a function of temperature plotted from equation (5) for 347 stainless steel with the MRI data plotted for comparison. The following table indicates the excellent agreement obtained:

PERCENT EXPANSION

347 Stainless Steel

Temp. (°K)	MRI	MSFC	Difference	% Error
250	0.079	0.080	.001	1.25
200	0.154	0.155	.001	0.65
150	0.218	0.220	.002	0.9
100	0.220	0.226	.006	2.17
50	0.297	0.306	.009	2.94

Both the MRI sample and the MSFC sample had a Rockwell B hardness of 84-87 prior to testing and 89-93 after testing.

With this run, the apparatus was considered to be fully qualified and calibrated, and the subsequent determinations on the other samples were made on this basis. These are shown in FIG 6 through 14, in graphic form, and in Tables 1 and 2.

The hysteresis effect is illustrated graphically in FIG 10 and 11. Two separate runs were made on a sample of 6061-T651 aluminum alloy. The sample was cooled to liquid helium temperature and then brought back to ambient temperature for a period of several days before the second run was made. The change in the linear coefficient of thermal expansion due to rearrangement of the grain structure during the heating-cooling cycle can be easily seen in both FIG 10 and 11.

Figure 12 illustrates the difference in expansion coefficient for two samples of the same aluminum alloy, 6061, differing only in heat

treatment. In general, the -T651 condition differs from the -T6 condition in being stress relieved. The difference in expansion coefficient for this relatively minor change is readily apparent.

Hardness determinations were made on all specimens after tests and were compared to nominal values for the material. The aluminum alloys decreased in hardness by about 20 percent, and the stainless steel alloys increased in hardness (indicating a partial transition from austenitic to martensitic) by a small percentage. Both results normally are expected for the particular materials involved and primarily serve as a warning against making successive determinations on the same material unless the material is heat treated to the original condition before the second run is made.

All specimens displayed the smooth curve expected for both coefficient of expansion and percent expansion. The data points, in almost all cases, lie completely on the curve; thus, an averaging or statistical reduction process was not required to produce the actual curve. Figure 16 shows a portion of the plot of length versus temperature for 321 stainless steel with the actual data points from the digital counter and illustrates graphically the extreme uniformity of the numerical data produced by this apparatus.

CONCLUSIONS

1. The apparatus is fully qualified and calibrated.

2. The apparatus is capable of determining the linear coefficient of thermal expansion in the range from 4° to 300°K with excellent accuracy.

3. Modifications to the apparatus have automated fully the fringe counting mechanism, increased the accuracy, and reduced operator time considerably.

REFERENCES

1. Dekker, A. J.: Solid State Physics. Prentice-Hall, Incorporated, 1962.

2. Cork, James M.: Heat. John Wiley and Sons, Incorporated, 1933.

3. Mott, N. F.; and Jones, H.: The Theory of the Properties of Metals and Alloys. Oxford, 1936.

4. Moeller, C. E.; and Rhodes, B. L.: Determination of Thermal Properties of Materials in Temperatures Ranging from -250 to 1,500°C. Contract NAS8-835, Progress Report No. 21, Midwest Research Institute, August 17, 1962.

5. Rhodes, B. L.; and Moeller, C. E.: Thermal Expansion of Several Technical Metals from 20 to 300°K. Presented at the 1962 Cryogenic Engineering Conference, University of California at Los Angeles, August 14-16, 1962.

6. Shenker, Henry, et al.: Reference Tables for Thermocouples. NBS Circular 561, U. S. Government Printing Office, 1955.

7. Moeller, C. E.; Rhodes, B. L.; Loser, J. B.; and Finch, H. L.: Determination of Thermal Properties of Materials in Temperatures Ranging from -250 to 1,500°C. Contract NAS8-835, Progress Report No. 18, Midwest Research Institute, May 17, 1962.

8. Merritt, George E.: The Interference Method of Measuring Thermal Expansion. Bureau of Standards Journal of Research, January - June 1933, pp. 59-76.

9. Saunders, J. B.: Improved Interferometric Procedure with Application to Expansion Measurements. Bureau of Standards Journal of Research, Vol. 23, July 1939, pp. 174-195.

10. Merritt, George E.: Application of the Interferometer to Measurements of the Thermal Dilation of Ceramic Materials. Bureau of Standards Bulletin, Vol. 19, 1923-1924, pp. 357-373.

TABLE 1

EXPERIMENTAL VALUES OF THE LINEAR COEFFICIENT OF THERMAL EXPANSION

10^{-6} CM/CM °K

Temperature, °K	321 SS	347 SS	355 SS	2017-T4 Al Alloy	6061-T6 Al Alloy	6061-T651 Al Alloy
12.5	0.15	0.07	0.19	0.15	0.35	0.47
37.5	1.52	0.59	0.74	1.78	2.76	3.63
62.5	5.52	4.89	4.38	6.76	7.46	7.47
87.5	8.64	7.89	5.64	10.79	11.73	10.47
112.5	10.66	0.98	6.78	14.22	13.78	12.88
137.5	11.84	11.76	7.49	16.27	15.48	15.20
162.5	12.90	13.14	8.28	18.24	17.37	17.17
187.5	13.84	13.92	8.48	19.46	18.82	18.43
212.5	14.66	14.70	9.61	20.71	20.19	19.64
237.5	15.04	15.21	10.08	21.25	21.24	21.13
262.5	15.52	15.52	10.71	21.83	21.78	22.22
287.5	16.06	16.22	11.10	23.59	22.72	22.92

TABLE 2

EXPERIMENTAL VALUES OF PERCENT EXPANSION

Temperature, °K	PERCENT EXPANSION						
	321 SS	347 SS	355 SS	2017-T4 Al Alloy	6061-T6 Al Alloy	6061-T6 Al Alloy	6061-T651 Al Alloy
0	-0.3155	-0.3093	-0.2098	-0.437	-0.4267	-0.433	-0.4217
25	-0.315	-0.309	-0.209	-0.436	-0.426	-0.432	-0.421
50	-0.311	-0.308	-0.207	-0.432	-0.419	-0.436	-0.411
75	-0.298	-0.295	-0.197	-0.415	-0.402	-0.407	-0.393
100	-0.276	-0.276	-0.182	-0.388	-0.376	-0.378	-0.367
125	-0.249	-0.251	-0.166	-0.353	-0.343	-0.343	-0.335
150	-0.220	-0.222	-0.147	-0.312	-0.303	-0.305	-0.303
175	-0.88	-0.188	-0.126	-0.267	-0.259	-0.262	-0.261
200	-0.153	-0.154	-0.104	-0.218	-0.213	-0.215	-0.215
225	-0.117	-0.117	-0.0797	-0.167	-0.163	-0.164	-0.166
250	-0.079	-0.079	-0.0545	-0.113	-0.111	-0.111	-0.113
275	-0.040	-0.040	-0.0278	-0.059	-0.057	-0.057	-0.0573
300	0	0	0	0	0	0	0
325	+0.040	+0.041					

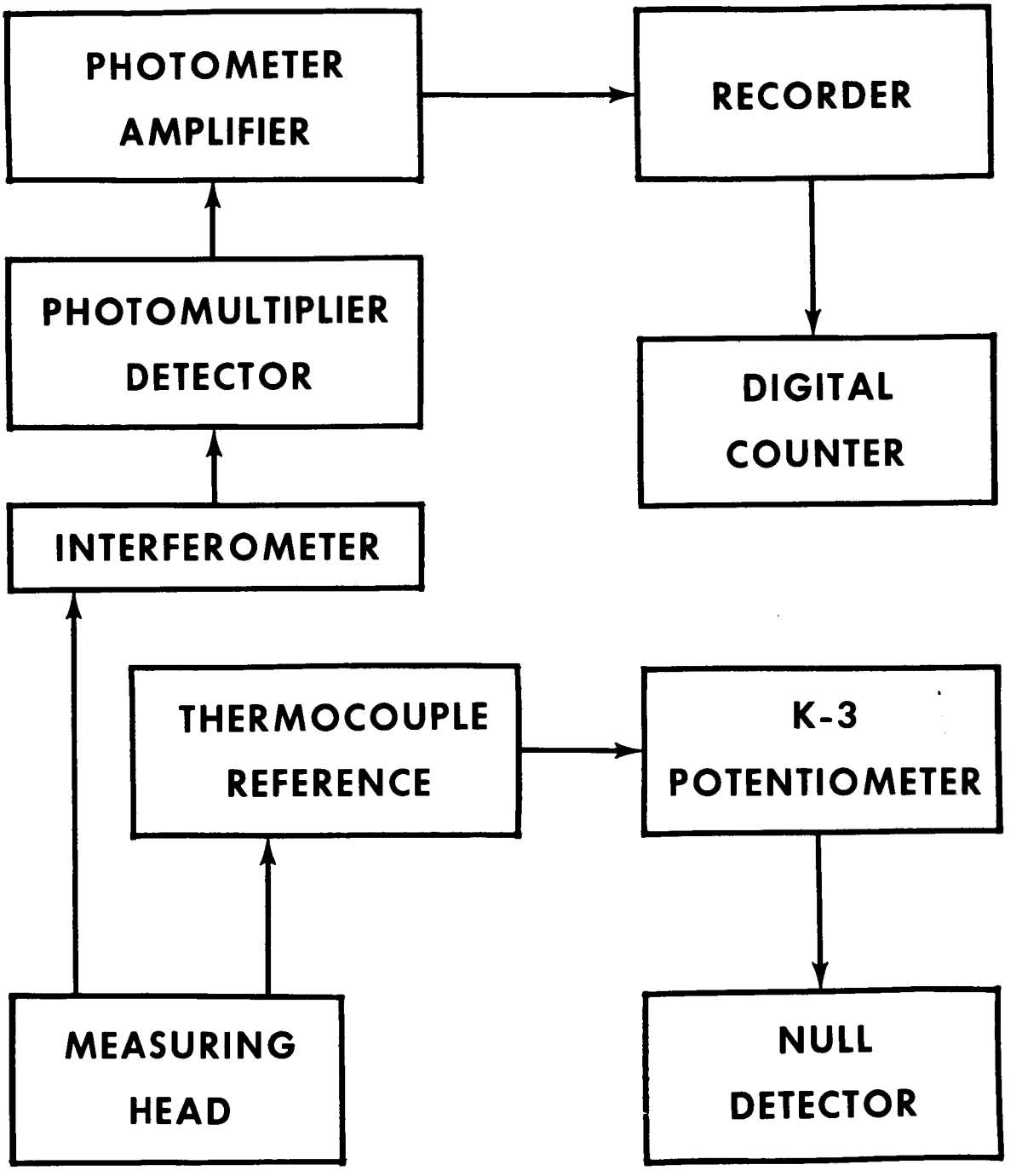

FIGURE 1. - MEASURING APPARATUS

(1) Retainer ring

(2) Leveling disc

(3) Leveling screws

(4) Adjustable light shield

(5) Sensing-rod guide tube

(6) Liquid helium dewar

(7) Liquid nitrogen dewar

(8) Fused-quartz dilatometer and sensing rod

(9) Guide ring

(10) Thermal expansion specimen

(11) Specimen heater

(12) Fused-quartz centering bead

(13) Upper Fizeau plate

(14) Tension screws

(15) Lower Fizeau plate

(16) Precision piston

(17) Precision cylinder

(18) Transfer tube

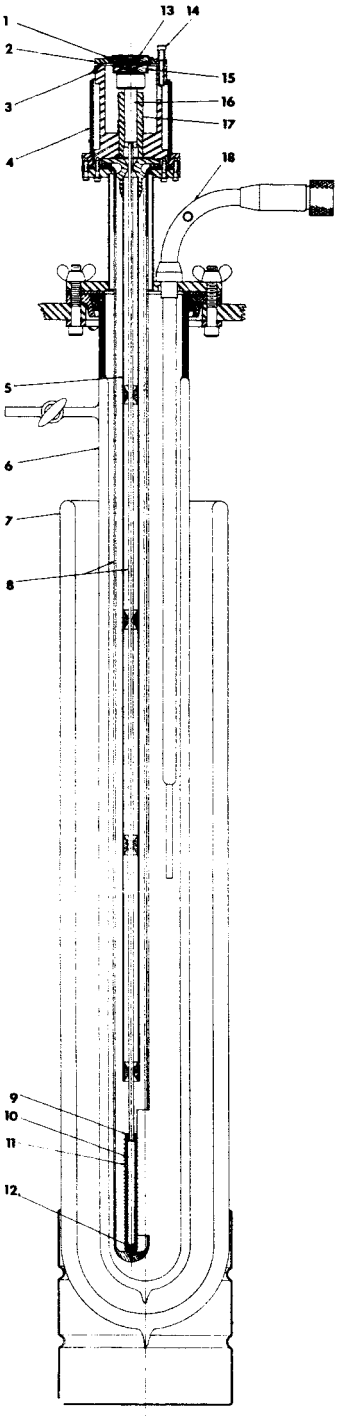

FIGURE 2. - INTERFEROMETER — DILATOMETER SYSTEM AND DEWAR ASSEMBLY

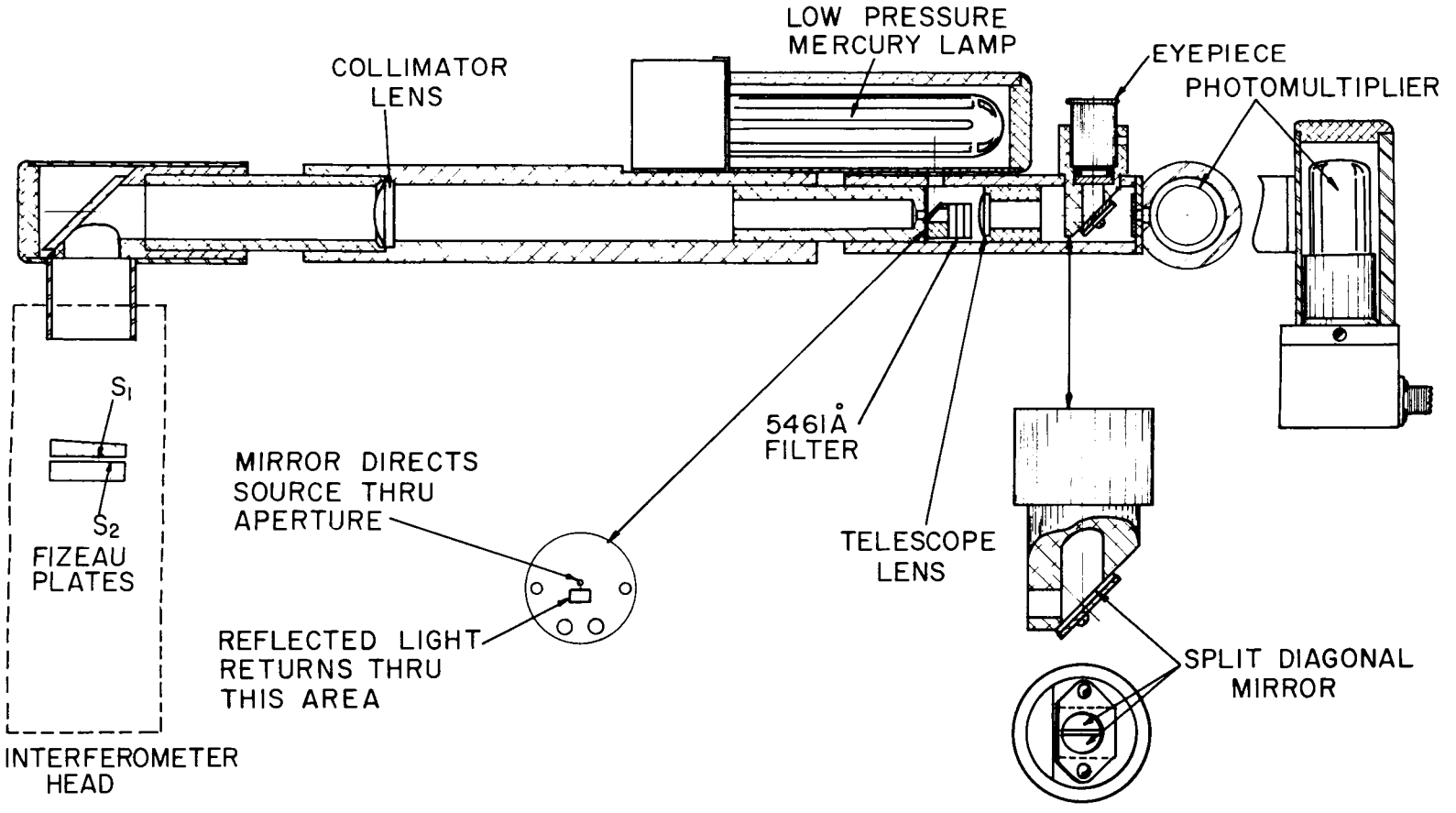

FIGURE 3. - INTERFEROMETER TELESCOPE AND FRINGE DETECTING COMPONENTS

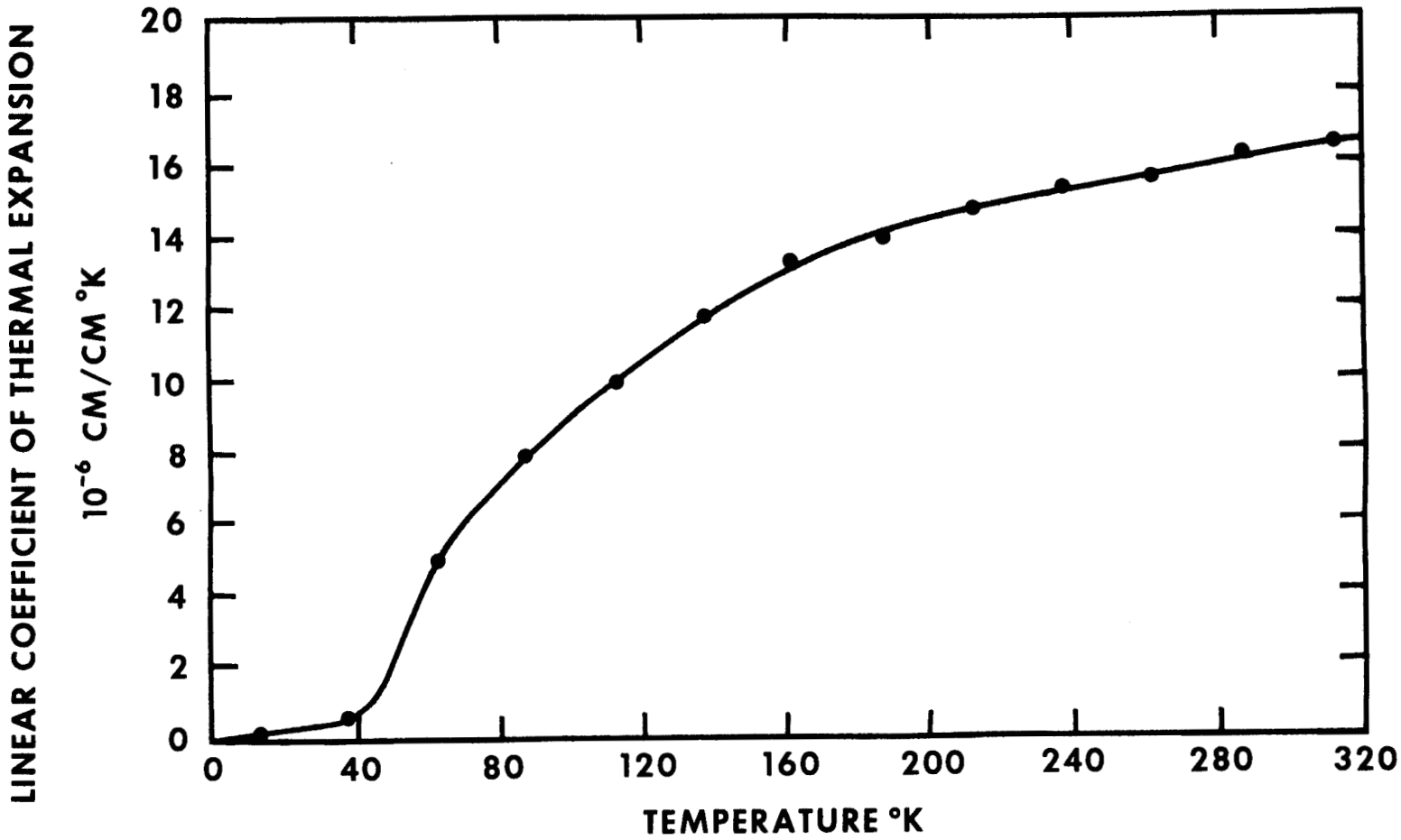

FIGURE 4. - LINEAR COEFFICIENT OF THERMAL EXPANSION,
347 STAINLESS STEEL

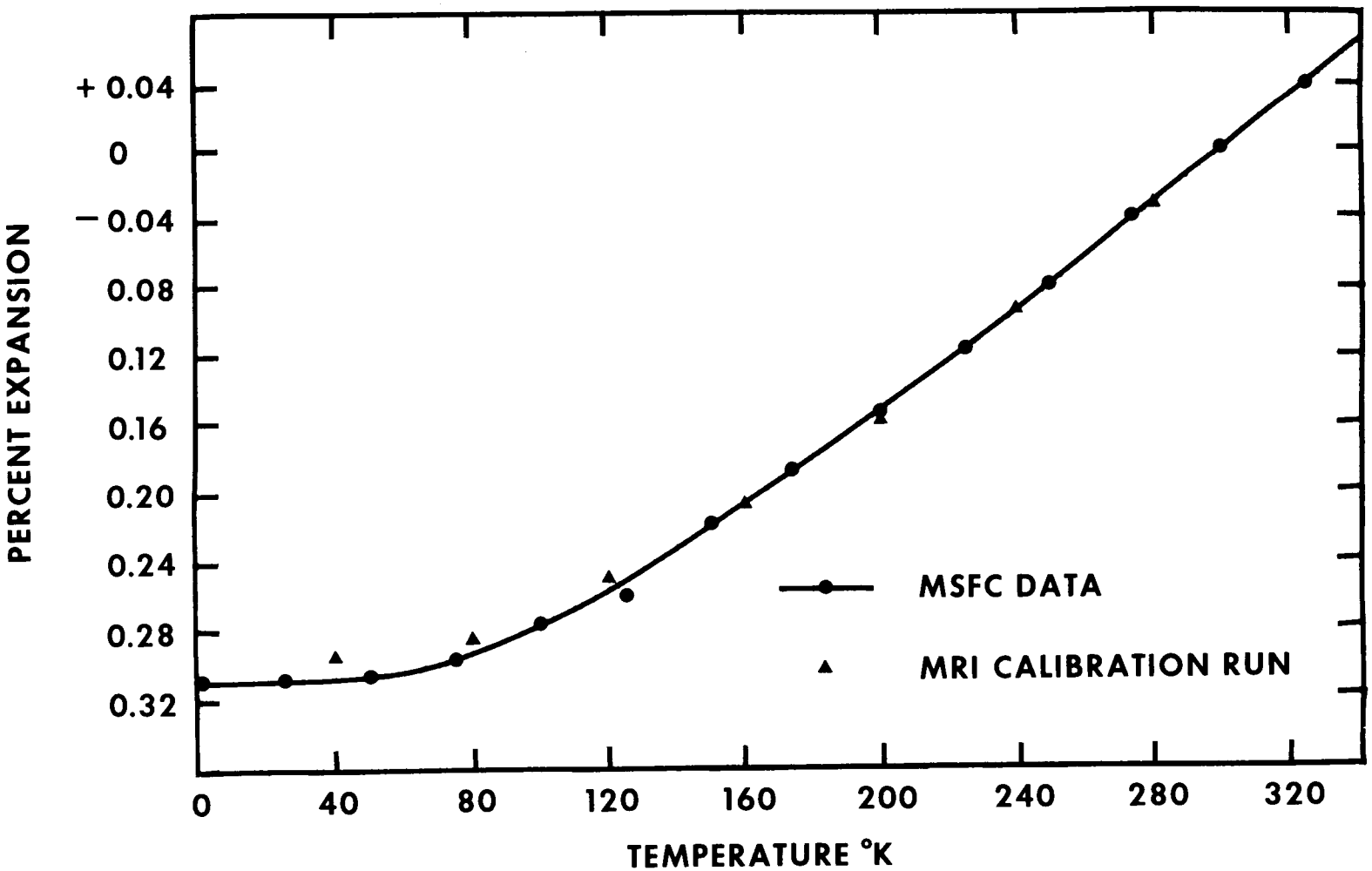

FIGURE 5. - PERCENT EXPANSION, 347 STAINLESS STEEL

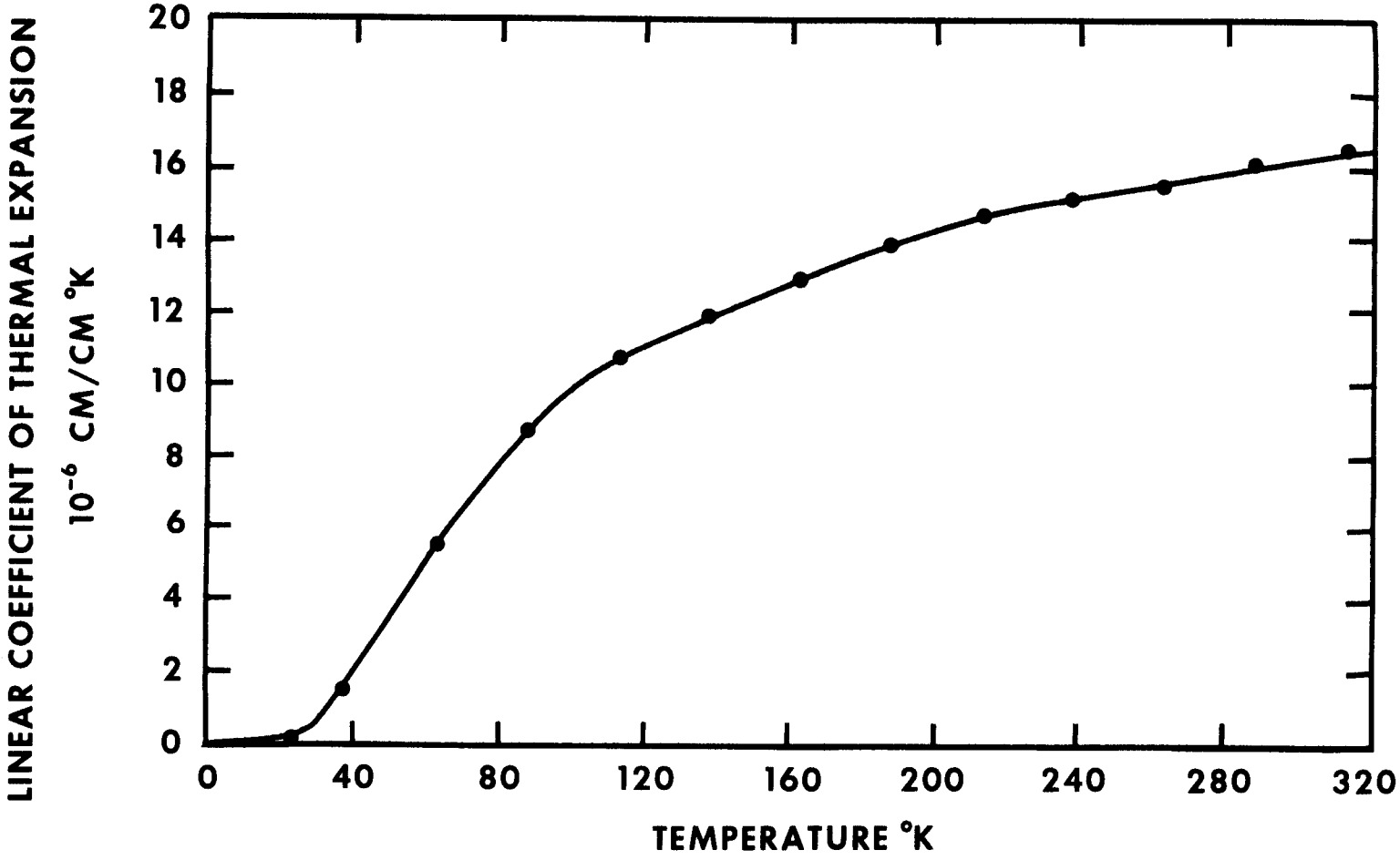

FIGURE 6. - LINEAR COEFFICIENT OF THERMAL EXPANSION, 321 STAINLESS STEEL

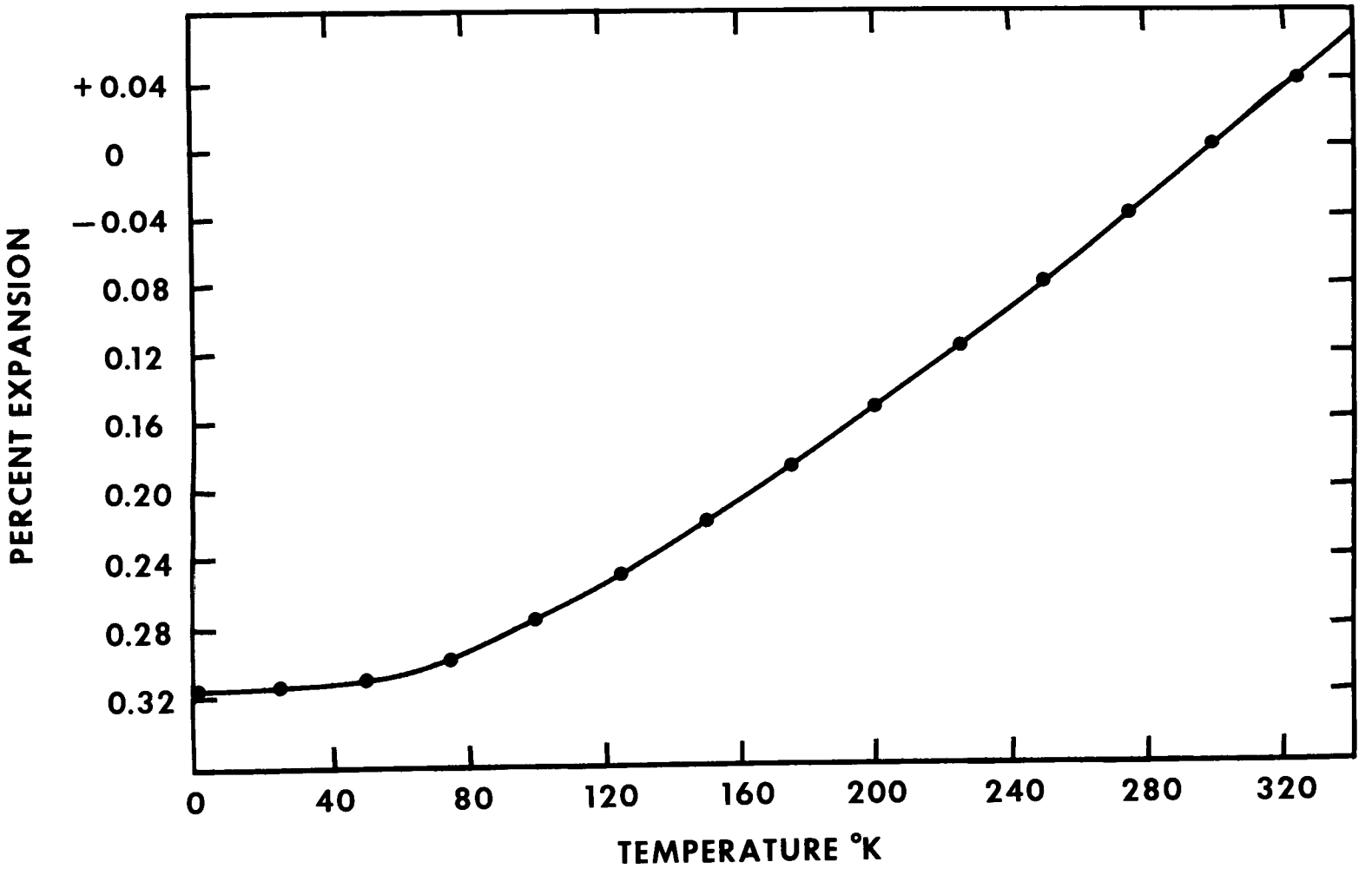

FIGURE 7. - PERCENT EXPANSION, 321 STAINLESS STEEL

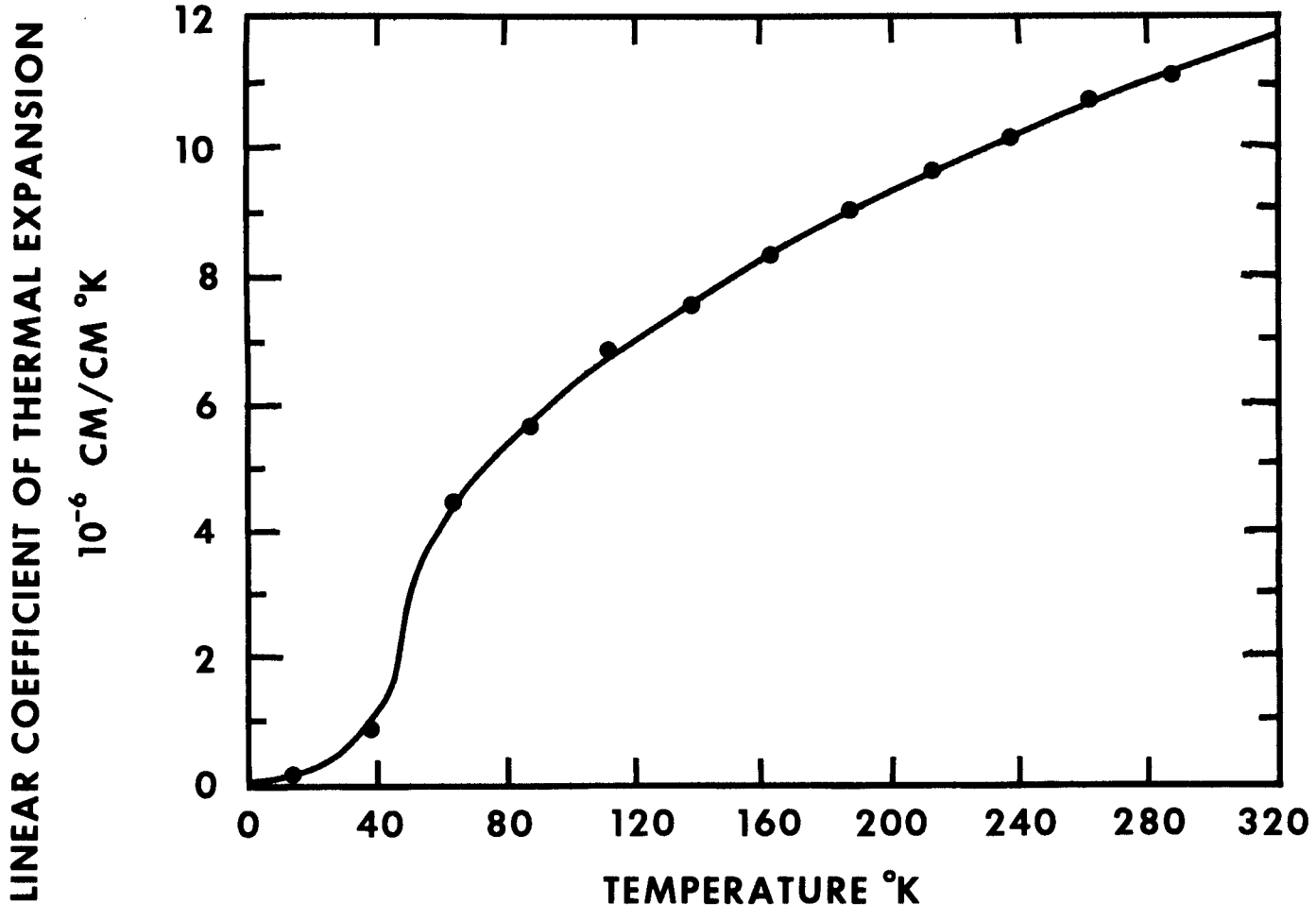

FIGURE 8. - LINEAR COEFFICIENT OF THERMAL EXPANSION, 355 STAINLESS STEEL

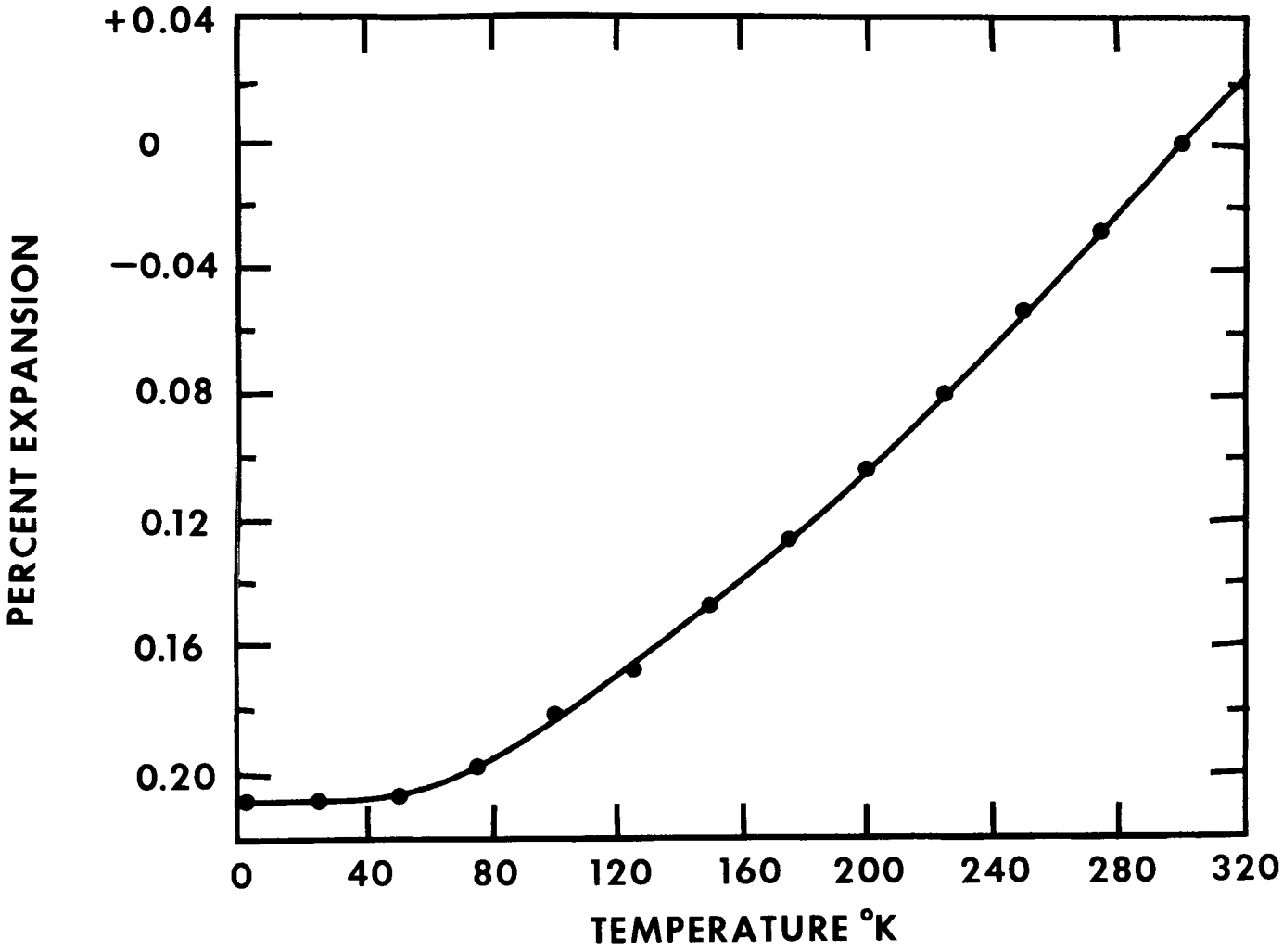

FIGURE 9. - PERCENT EXPANSION, 355 STAINLESS STEEL

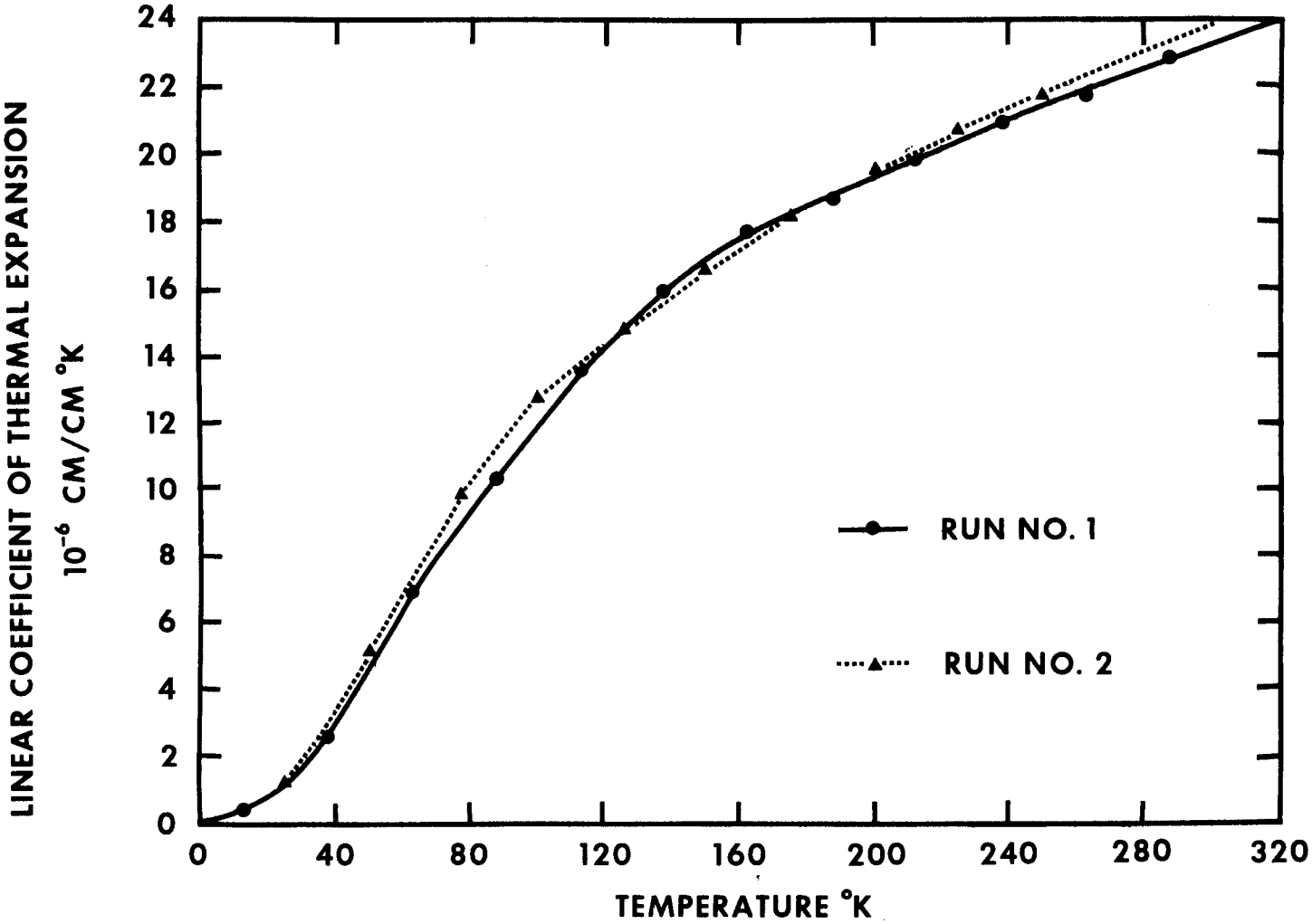

FIGURE 10. - LINEAR COEFFICIENT OF THERMAL EXPANSION, 6061-T6 ALUMINUM ALLOY

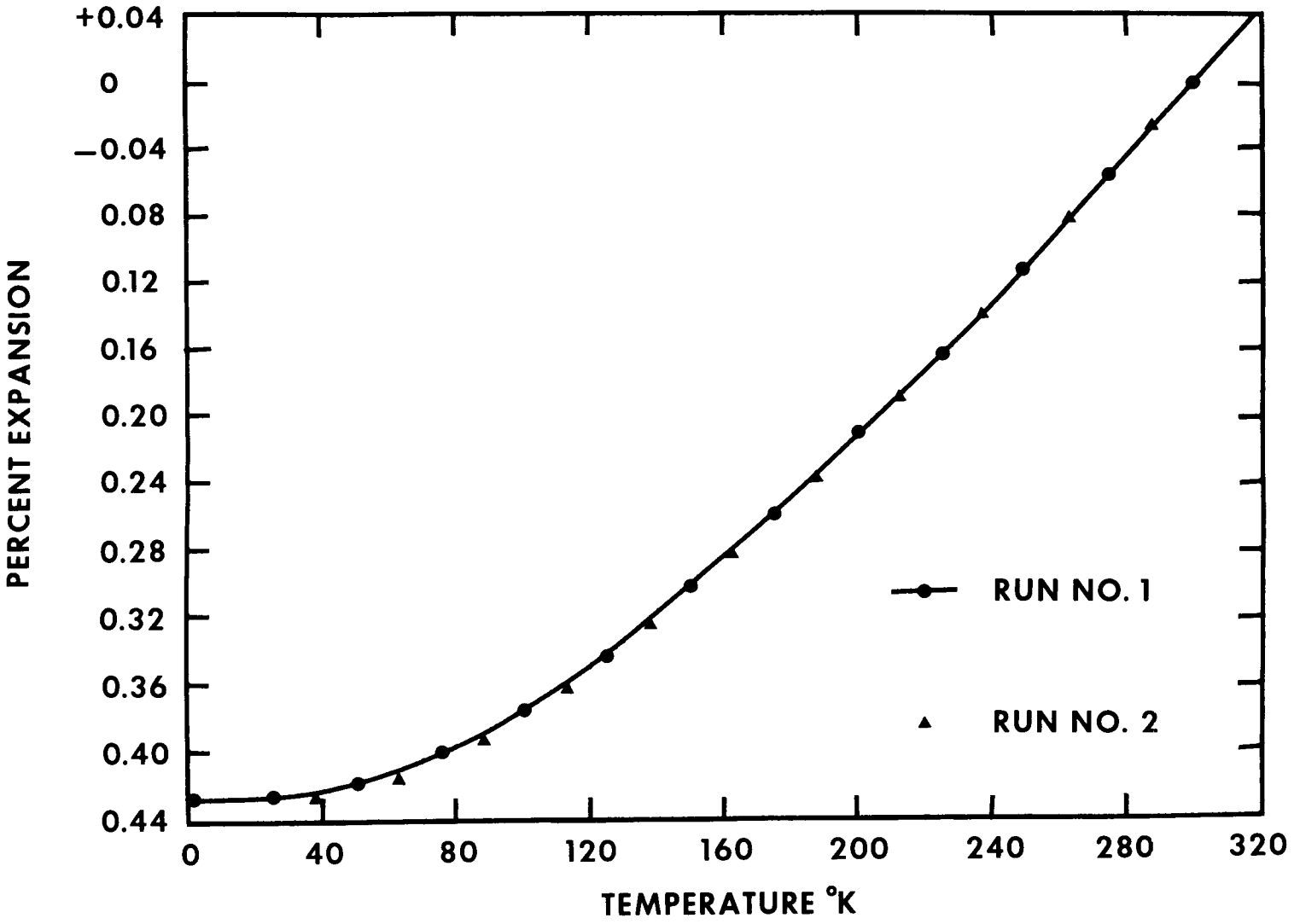

FIGURE 11. - PERCENT EXPANSION, 6061-T6 ALUMINUM ALLOY

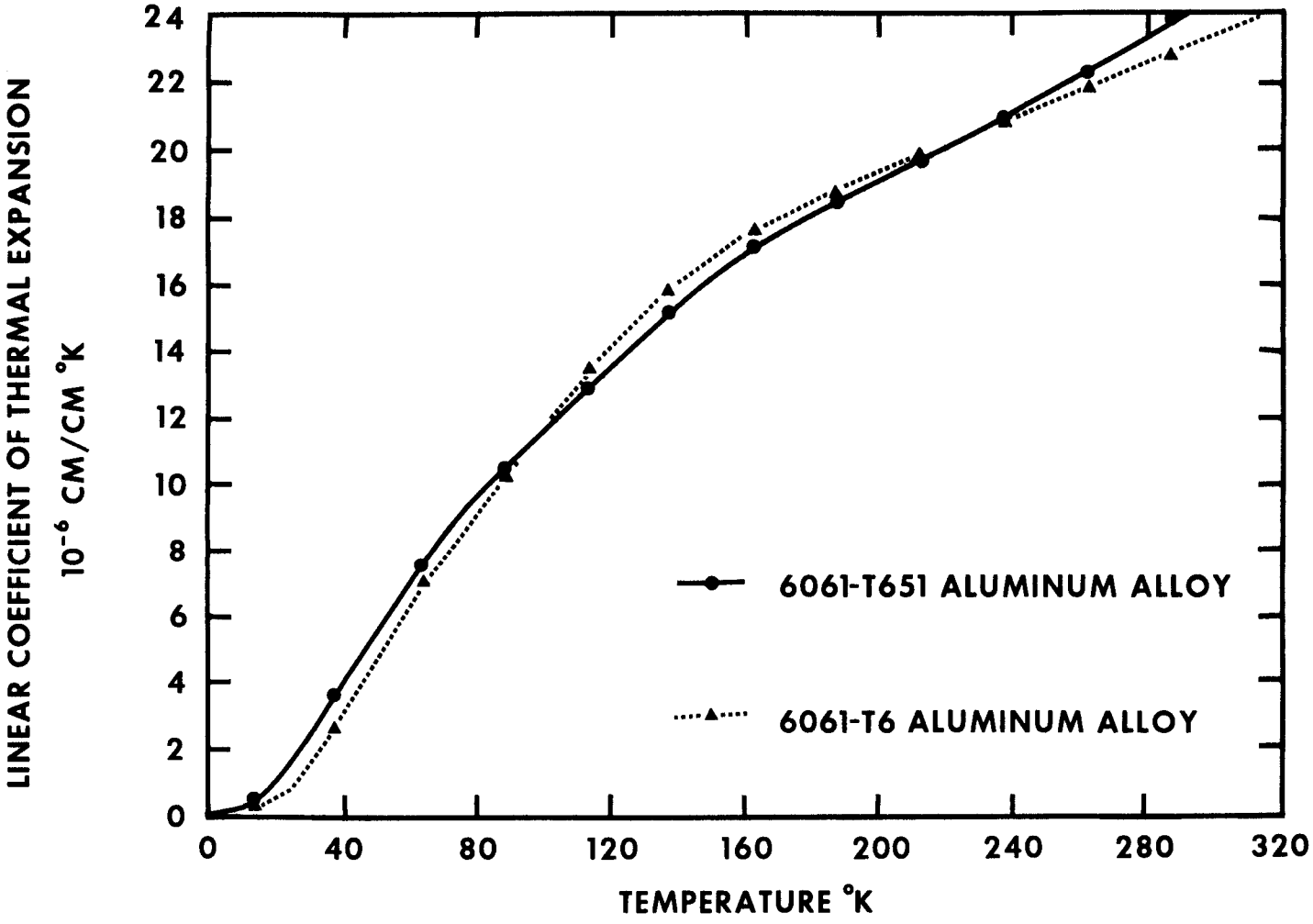

FIGURE 12. - LINEAR COEFFICIENT OF THERMAL EXPANSION,
6061 - T651 AND 6061 - T6 ALUMINUM ALLOY

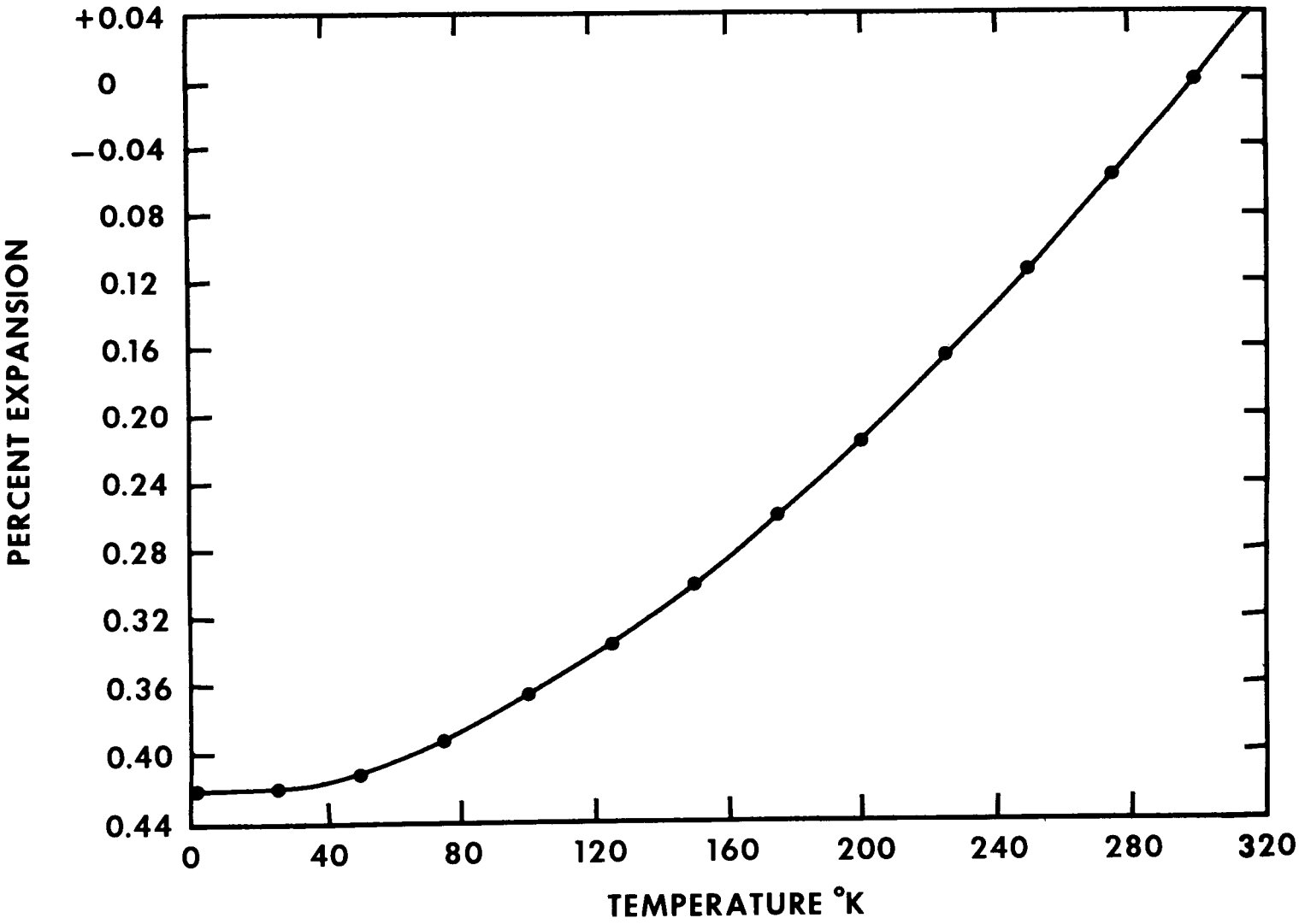

FIGURE 13. - PERCENT EXPANSION, 6061-T651 ALUMINUM ALLOY

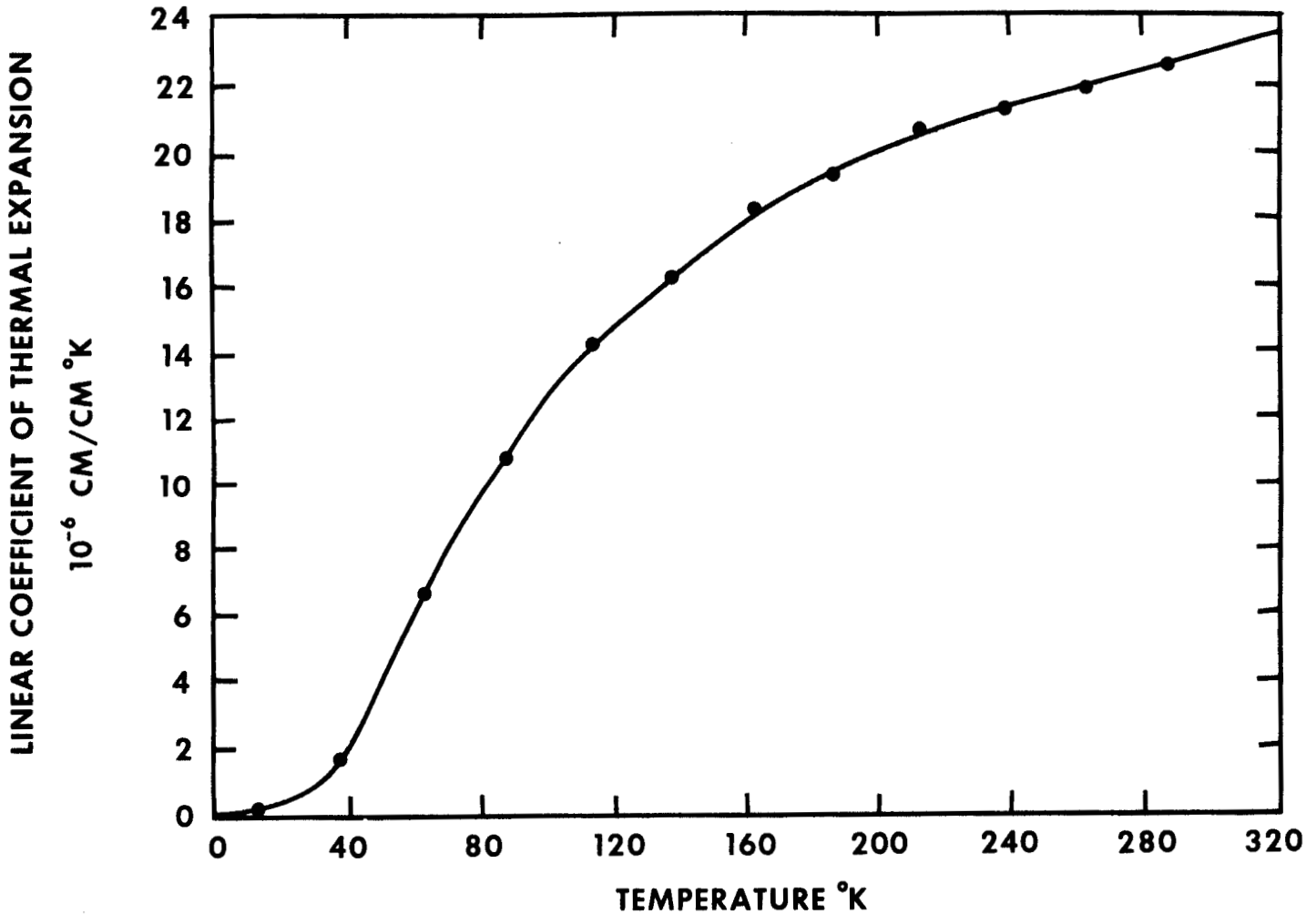

FIGURE 14. - LINEAR COEFFICIENT OF THERMAL EXPANSION, 2017-T4 ALUMINUM ALLOY

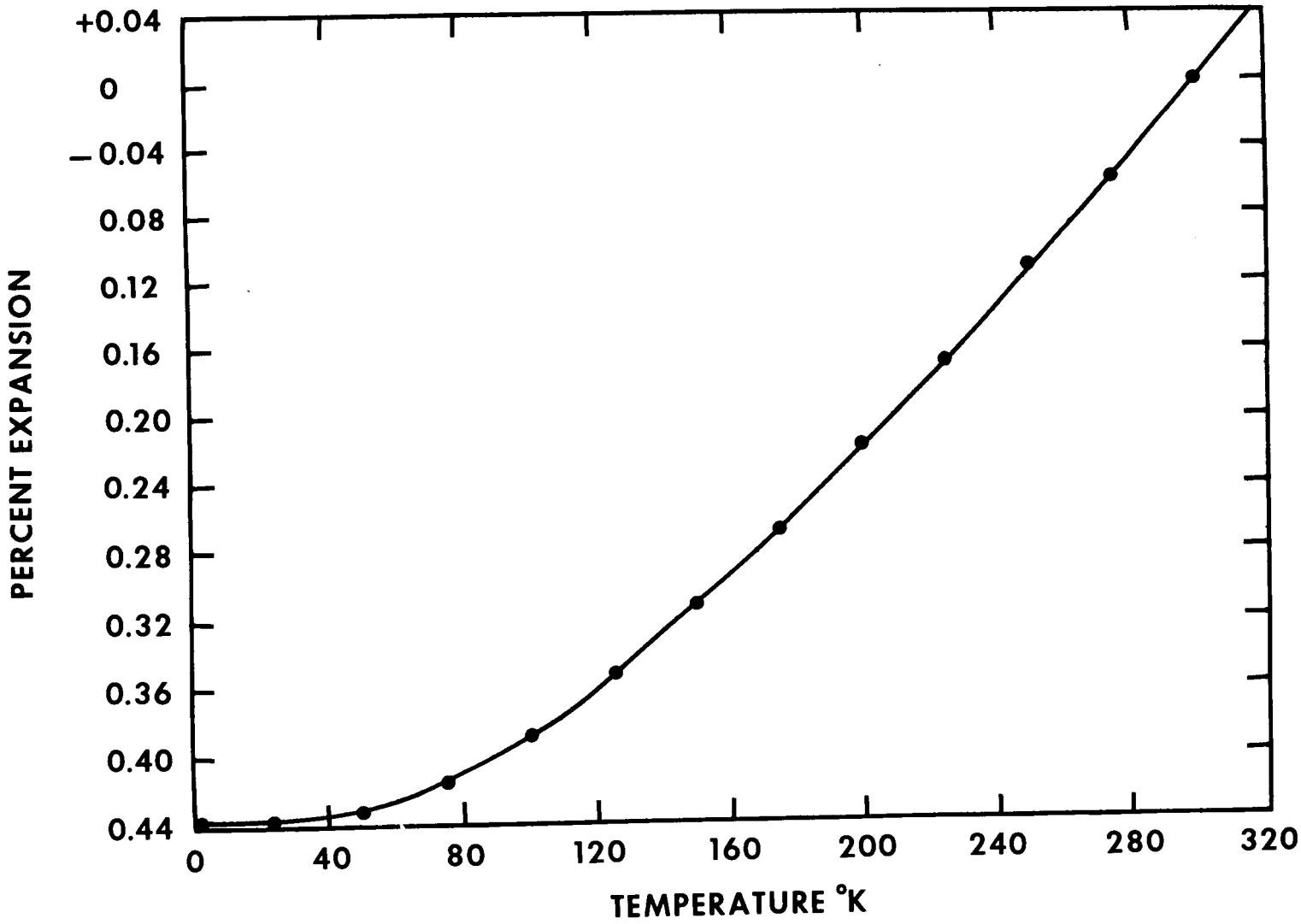

FIGURE 15. - PERCENT EXPANSION, 2017-T4 ALUMINUM ALLOY

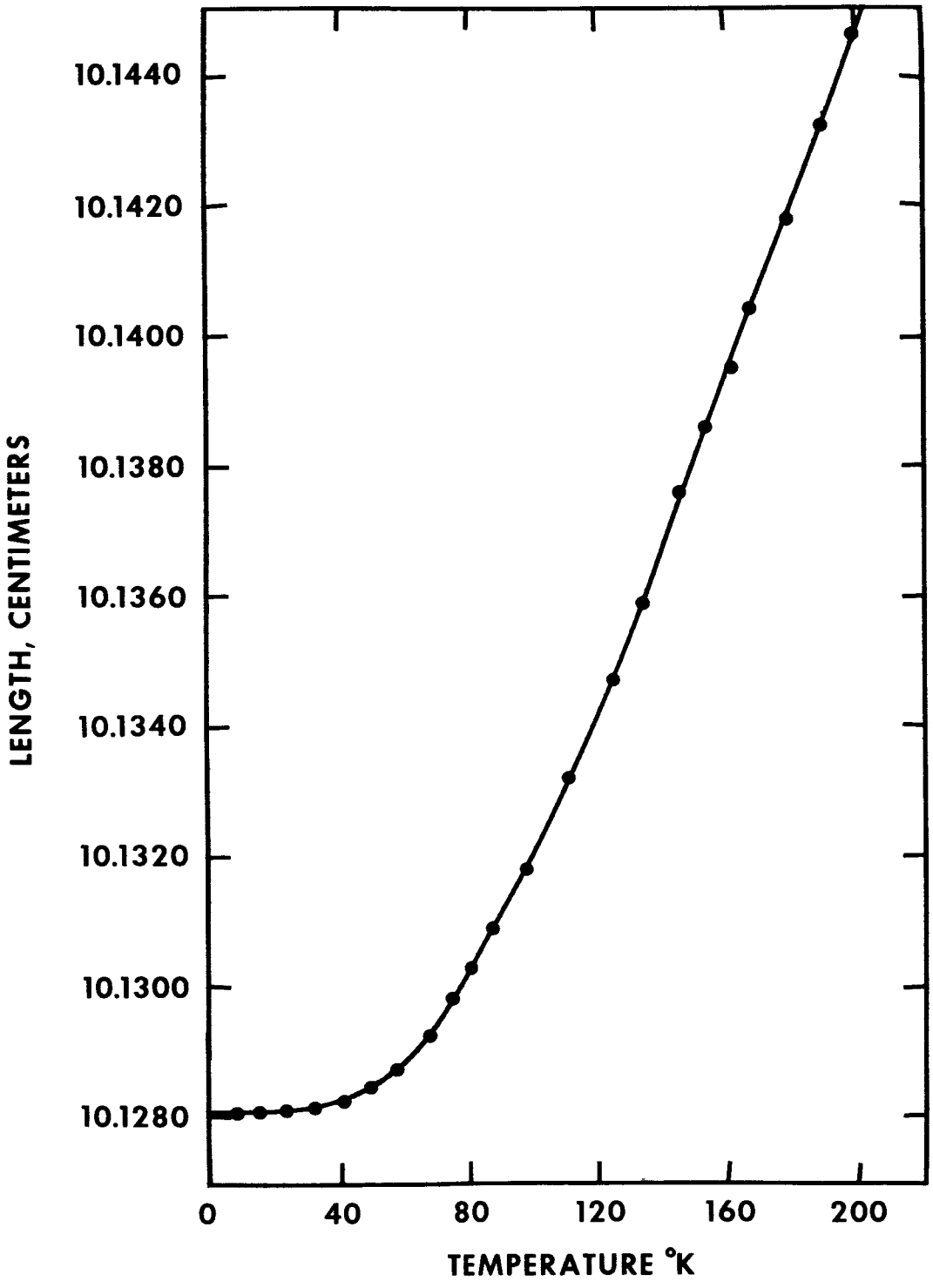

FIGURE 16. - LENGTH VS TEMPERATURE, 321 STAINLESS STEEL

LOW TEMPERATURE THERMAL EXPANSION OF STRUCTURAL METALS

By J. C. Horton, C. F. Smith, and R. C. Ruff

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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